HERRI.001APC

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PROCEDURE FOR THE MANUFACTURE OF HIGH CONCENTRATION MANGANESE MINITABLETS FOR ALUMINIUM BATH ALLOYING, AND THE DEVICE FOR EXECUTING IT

DESCRIPTION

OBJECT OF THE INVENTION

The present invention refers to a procedure for the manufacture of high concentration manganese (Mn) minitablets for aluminium (Al) bath alloying, the purpose of which is to produce Mn minitablets with a 90-98% concentration of this metal, for adding in Al smelting.

The object of the invention is to produce a minitablet product composed of Mn and Al powder whose first component is obtained by electrolysis and grinding, while the second component is an atomised powder produced by means of mechanical processes, both components being them mixed and compacted to form minitablets with a high Mn concentration.

A further object of the invention is the device for the execution of the above-mentioned procedure, the device where the loading, dispensing, compacting and final forming of the minitablets take place.

BACKGROUND OF THE INVENTION

The alloying of aluminium baths with manganese has changed substantially in recent times, and from the original addition of lumps of metal, which gave rise to serious problems of purity and dissolving rate, there has been a shift towards two different concepts of alloying: on the one hand, the use of parent alloys, consisting of Al and Mn manganese alloys with a 10 to 25% Mn content, and, on the other, the addition of powdered Mn by means of injecting the powder into the furnace. Although both methods are still employed today, their use has declined drastically since the first compact Mn pellets were introduced towards the end of the seventies. These pellets, which came in the form of tablets, minitablets or briquettes, combine concepts of the two previous methods,

take advantage of their strong points and reduce their drawbacks. The pellets consist of Mn powder in a concentration usually above 75% compacted using Al powder as the binding agent, a flux, or a mixture of both, in a concentration of up to 25%. These materials substantially reduced the amount of cold material which is added to the Al furnace in the alloying operation in comparison with parent alloys. Furthermore, parent alloys usually contain 75 to 90% second smelting aluminium, which could give rise to problems in the molten metal, besides calling for a stock 4 times higher than that of compacted powder alloying agents. Moreover, they are easy-to-use materials that do not require the investment in equipment and safety that is necessary for powder injection.

The great financial step that was taken on changing parent alloys containing a maximum of 25% Mn to compact alloying agents whose Mn content is 75% or more has generated constant pressure on the manufacturers of compact alloying agents to obtain materials that, while being effective in the Al bath alloying process, also succeed in increasing the Mn concentration in the compact alloying agent. In this respect, no materials are available on the market that contain a percentage above 85% Mn, due mainly to the problems of compactibility of Mn, an abrasive and non-ductile material. In addition, it is suspected that the material may not dissolve as quickly as the compact materials of lower Mn concentration due to the reduced proportion of Al and/or flux, which also act as disintegrators of the compact when this is put into the furnace, as reported by the scientific literature on the subject.

As the active alloying element of the compacts is Mn, the decreased Al content brings a series of advantages for the founder. The amount of material to be added to the furnace is smaller, which means that less cold material is added to the Al bath and that raw material stocks are reduced. Similarly, there is a cut in material transport costs, which will be significantly lower than those of 75% or 80% compacts. Besides this, the price of products depends less on the value of Al, subject to the changes in its quotation on the London Metal Exchange, and since Al is currently more expensive than Mn, the cost of the set of the raw materials used in production would also be lower. Lastly, we have to consider that the founder/user is not interested in adding a material (Al powder) to his furnace that he is able to sell himself and which, moreover, has a value

added due to atomisation, which is lost when smelting it again.

Despite these financial advantages, no compact Mn materials have appeared on the market with a concentration of 90% or more. The attainment of this objective raises a series of scientific challenges when it comes to flow production of these materials. On the one hand, experience indicates that the pressing process has to be improved in order to be able to reach these Mn percentages. On the other, the raw materials have a series of factors that may be modified when it comes to achieving a better performance. In addition, it has to be confirmed whether it is really necessary to have compacts in the furnace with Al powder concentrations of more than 10% or 15% for the Mn dissolving rate to be acceptable, or whether compacts with less than 10% Al dissolve in the furnace at a suitable rate.

The present study concentrates on the flow production and performance in the Al furnace of alloying minitablets (cylindrical in shape) containing Mn in a concentration of more than 90%, Al being the remaining material. Although it would be desirable to have this concentration available in standard sized tablets as well, the need to apply high pressures to the material means that the study is complicated if the size of the compact diameter is larger than 40 mm. On the other hand, fluxes were initially rejected in this study insofar as they are materials whose binding action is considerably inferior to that of Al powder.

With regard to the raw materials to be used, Mn is the first limitation of the study. The chemical requirements of Al baths involve the use of Mn of a high chemical purity, usually above the 99.7% level, which can only be assured if the Mn is produced by electrolysis. At present electrolytic Mn is only produced in the Republic of South Africa and the People's Republic of China, which reduces the possibilities of finding materials with different specifications. Mn, which is usually in flake form, has to be converted into powder by grinding. The material normally used in the compacting of Mn minitablets has a grain size of less than 450 microns. Mn powder is highly abrasive, a property that is enhanced if the amount of fines (powder below 100 microns) increases, and which has a direct effect on the pressing quality and the average life of the materials (punches and liner) of

the press in which the material is compacted.

The situation is very different with regard to the Al powder involved. There is a great variety of Al powders on the market that may be used in continuous industrial processes and with different applications. In the case of compacting Mn half-tablets, it is normal to use Al fractions above 100 microns and below 1000 microns insofar as grain size is concerned. These fractions are the ones that Al producers generally regard as a by-product in their production processes, inasmuch as the fine fractions of Al (below 100 microns) are the ones that have valuable applications in aeronautics and pyrotechnics on account of the explosive property of Al. This fact means that again the production of a material of specific characteristics for the compacting of Mn tablets is skewed or subject to production conditions independent of the application that this study sets out to examine.

In general, the Al used in the production of Mn minitablets is a gas-atomised powder, although materials may be used that are obtained by mechanised atomisation procedures, annealed materials or micronised swarf. As a rule, atomised powders are the most suited to the requirements of the main functions of Al.

In the production of the Mn minitablets, Al acts as a binding agent, whereas electrolytic Mn, being highly abrasive and non-ductile, is a material that does not compact on its own. Potential improvements in the process apparently lie in the application of higher pressures to the material so as to enable these materials to be compacted. Apart from using higher performance hydraulic units and applying greater force to the pressing punches, another possibility is to reduce the diameter of the minitablets, as the smaller the area of application is, the greater the actual pressure. This represents a problem at industrial level, as smaller diameter minitablets give rise to lower productivity (minitablets weigh less). To overcome this problem, it is necessary to work with several punches at the same time, and the pressing process has to be effective for all the minitablets made in a cycle. This means that all the liners have to be filled properly with the material to be compacted, that this must be mixed properly and not be different in each of the liners in which it is received, and that the material must flow smoothly

to these liners. In this respect, it is extremely important to stop the mixture of Mn and Al powders from becoming separated at any time in the process (a problem that could arise easily since the two materials have widely differing densities) and, furthermore, that the equipment should be suitably sized so as to apply the pressure needed for compacting.

DESCRIPTION OF THE INVENTION

The procedure that is advocated offers a solution to the problems and difficulties mentioned in the previous section, for which purpose it is specified that, starting from the two components used, which have to be mixed, namely Mn and Al, Mn minitablets with a concentration of more than 90% should be compacted by using Mn produced by electrolysis and ground from flakes of Mn of a chemical purity of 99.7% or more, which is subjected to a screening process with a sieve with a mesh of less than 450 microns; the special feature of the Mn grinding process is that it is controlled so that the content of fine Mn powder, with a size of less than 100 microns, should not be more than 15%, as above this proportion the compacting of Mn minitablets cannot be assured with over 90% Mn in their composition.

The procedure also includes the fact that the most suitable Al for successfully compacting Mn minitablets is atomised powder, which is produced by mechanical processes, with controlled size distribution, its nominal grain size intervals being between 100 and 800 microns, with over 80% of the powder in the 350-720 micron range.

This grain size distribution is coarse enough to enable the material to be compacted and fine enough not to retard the dissolving rate, through having reduced the number of Al grains with the increased Mn concentration in the minitablet.

The invention also refers to the device for executing the foregoing procedure, consisting of a hopper for the reception of an Mn and Al mix with the afore-mentioned characteristics, there being a central product diffuser in this hopper which forces the product to flow through the sides of the hopper to

prevent the mix directly reaching the feeder of a second hopper which discharges into the respective pressing or compacting chamber, where pressing punches will come into action.

The device has appropriate means that enable maximum, minimum and safety levels to be kept under control in the compacting chamber so that it remains at a level of filling all the time such that none of the punches may try and make an off-load compacting stroke.

As one of its main innovative features, besides the afore-mentioned central diffuser, the device includes a honeycomb dispensing valve interposed between the feed hopper and the compacting chamber, which is provided with a series of dies that are mounted on a support integral with the actual feed hopper, so that the support-hopper assembly is able to run along guides, in either direction, under the action of a pneumatic device, on which guides there is in turn a moving punch support mounted, also driven by a pneumatic ram, so that the support-hopper movement is independent of the moving punch movement, although such movements must be synchronised in order to fill, press, compact and eject the formed minitablet.

Besides the aforesaid central diffuser and the location and use of the honeycomb dispensing valve, as an innovative feature, the device also includes three electrical control means to monitor the maximum, minimum and safety levels, corresponding to compacting chamber filling.

DESCRIPTION OF THE DRAWINGS

To supplement the description being given and in order to assist a better understanding of the features of the invention, in accordance with a preferred example of a practical embodiment of same, as an integral part of this description a set of drawings is adjoined, wherein, for purely illustrative and non-restrictive purposes, the following is represented:

Figure 1.- It shows the graph corresponding to the standard grain size distribution of the Mn used in the invention procedure. The *y*-axis contains grain

size intervals in millimetres, and the *x*-axis the percentage by volume of each fraction. Grain size was measured by laser diffraction with dry method sample insertion.

Figure 2.- It shows a representation referring to the micrograph of the Al powder in granules used in the invention procedure.

Figure 3.- It shows the graph referring to the standard grain size distribution of the Al used in the invention procedure. The *y*-axis contains the grain size intervals in millimetres, and the *x*-axis the percentage by weight of each fraction. Grain size was measured by a sieve tower.

Figure 4.- It shows a diagrammatic, partially sectional, side elevational view of the device for the execution of the invention procedure.

Figure 5.- It shows an elevational view, front and sectional in this case, of the same device as in the previous figure.

PREFERRED EMBODIMENT OF THE INVENTION

The invention procedure, designed to produce Mn minitablets by compacting, with a concentration of more than 90% of this metal, is based on using electrolytic Mn ground from flakes of a chemical purity of 99.7% or more. The product is then screened with a sieve with a mesh of less than 450 microns, since it has been found that materials containing significant fractions of a larger grain size give rise to much lower dissolving rates in the aluminium furnace. The grinding process is controlled so that the content of Mn fine powders (below 100 microns) is more than 15%, as above this percentage it has been found that the compacting of minitablets cannot be assured with more than 90% Mn in its composition. Figure 1 shows the graph referring to the standard grain size distribution of the Mn used.

The tests made indicate that the Al powder most suited for compacting Mn tablets with a concentration of more than 90% is powder atomised by mechanical procedures, the special performance of this AL powder being due to

its spongy granule structure that permits suitable fluidity on the metal surfaces of hoppers but which maintains sufficient air holes in the grains, so that the material is endowed with greater compressibility. Figure 2 shows the micrograph of the Al powder in grains, according to a microscope enlargement of this type of powder.

The foregoing Al powder also has a controlled grain size distribution, its nominal grain size intervals being between 100 and 800 microns, with over 80% powder between 350 and 720 microns. This grain size distribution is coarse enough to enable the material to be compacted and fine enough not to retard the dissolving rate, through having reduced the number of Al grains (which trigger the dissolving reaction on the minitablet Mn in the furnace) with the increased Mn concentration in the minitablet. Figure 3 shows the graph referring to the standard Al grain size distribution in the grains used.

The device for executing the procedure is represented in figures 4 and 5, comprising a hopper (1) for reception and storage of the mix, which is fed in through the respective filler neck (2), a mix which, as stated, is composed of Mn and Al. The mix has to be homogeneous and, on being received in the hopper (1), it falls on a centrally positioned diffuser (3), a diffuser (3) that has a conical layout and is supported on legs (4), so that this diffuser forces the product to flow through the sides of the hopper (1) and never directly onto the feeder hopper (5) provided at the outlet of the hopper (1), and from which hopper (5) the product moves onto the compacting hopper (6). The diffuser (3) prevents the effects of product separation and assures continuous fluidity at the same level of product in the hopper (1). The compacting hopper (6) is a vertical continuation of the feeder hopper (5), so that the former defines a chamber which maintains a product level and in which the compacting is done by means of both fixed punches (7) and moving punches (8).

The compacting hopper (6) is provided with a series of dies (9), of varying number depending on the size of the device, and the product or Mn and Al powder reaches these dies (9) by way of a honeycomb valve (10) interposed between the feeder hopper (5) and the compacting hopper (6), so that a metered amount of product passes through this valve and is loaded onto each one of the dies (9), as the honeycomb valve (10) forms a sort of drum-sector that is loaded

with a given quantity of product so that, when this valve turns through an angle, the corresponding sector load discharges on the compacting hopper (6) and the product reaches the respective die (9). The dies are arrayed on a support (11) which is integral with the actual compacting hopper (6), and that support-hopper assembly is mounted on guides (12), along which it may move in either direction under the action of a pneumatic device, on which guides (12) there is in turn a moving punch (8) support (13) mounted, also driven by a pneumatic ram or device. The support-hopper movement is independent of the moving punch movement, although such movements must be synchronised in order to fill, press, compact and eject the formed minitablet.

The fixed punches (7) are arranged co-axially facing the moving punches (8), the latter being installed on a static support (14).

In this way, when the support (11) with the compacting hopper (6) moves forward, the die (9) is filled and then trips the moving punch (8), which advances and compacts the material located between it and the fixed punch (7). The moving punch (8) then moves back and the support-hopper assembly slides slightly forward so that the fixed punch (7) ejects the minitablet, whereupon the cycle starts over again.

It is essential for this device to maintain a minimum product column level in the compacting chamber (6), so that none of the punches attempts to compact an empty die, which would result in the breakage of the punches and column or chamber. This level is maintained by the use of three electrical controls and the afore-mentioned honeycomb valve (10), controls which correspond to references A, B and S, and which indicate the maximum level, minimum level and safety level of the product in the compacting chamber (6), all of this in such a way that the safety level S causes the device to shut off if the product drops below this level because there will be a risk of emptying the chamber, whereas level B is the product level that permits a reproducible column weight to be maintained capable of assuring suitable fluidity and consistent reproducible filling at all the punches. When the product has reached that level, the honeycomb valve (10) opens and dispenses more product from the hopper. This honeycomb valve (10) closes when the product reaches the maximum level A.

To obtain proper compacting of the half-tablet with an Mn concentration of 90% or more, it is necessary to work with punches capable of applying a pressure of 7500 Kg/cm^2 of punch. In a practical example a check was made on the mechanical strength of the product obtained with 90% and 95% Mn, in the conditions explained, a mechanical strength check that was carried out by means of a drop test consisting of dropping a number of minitablets onto a cement floor from a height of 1 m, recording the number of impacts required to cause breakage and for the loss of 2% weight of the minitablet.

	Minis Mn 90%	Minis Mn 95%
Number of tests	5	5
Drops to 2% weight loss	3 ± 1	1.3 ± 0.6
Drops to breakage	3.7 ± 0.6	2.3 ± 0.3

Dissolving tests of these Mn minitablets with concentrations of 90% or more were conducted in Al baths, using for this purpose a rotary gas-fired semi-industrial furnace with a capacity of 400 kg Al. The experiments were performed in accordance with regular standard processes for the addition of minitablets, bath slag removal, stirring and sample collection. The samples were analysed by spark spectrophotometry.